

PATENT ABSTRACTS OF JAPAN

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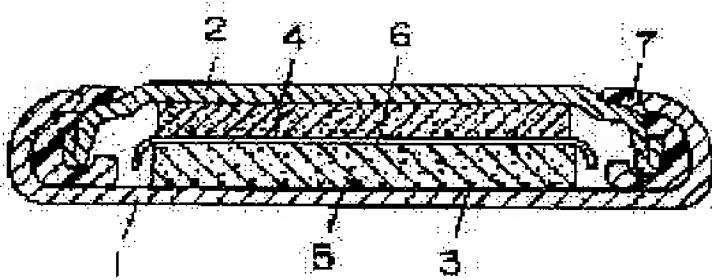
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(54) NONAQUEOUS ELECTROLYTE SECONDARY BATTERY

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a high energy density battery which is operable in high electric potential by forming a positive electrode active material of a metallic iodate compound.

SOLUTION: In a positive electrode 5, an iodate compound or a metallic iodate compound containing one or more kinds from among Fe, Co, Cu, Zr and Ag is used as a positive electrode active material, and when manufacturing a pole plate, a mix by mixing 10 pts.wt. of polyvinylidene fluoride of a binding agent and 5 pts.wt. of acetylene black of a conductive agent to 85 pts.wt. of either of various metallic iodate compounds, is molded on a current collector 3. Such various iodate compounds function as a positive electrode material of a nonaqueous electrolyte secondary battery, and particularly, the metallic iodate compounds containing one or more kinds from among Fe, Co and Cu are high electric potential, and are also superior in capacitive density. When this positive electrode material is used for the nonaqueous electrolyte secondary battery, nitride, oxide or an alloy containing lithium metal and lithium can be used as a negative electrode active material.



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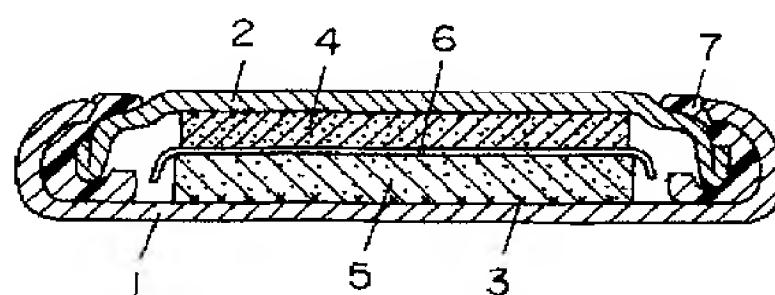
(54)【発明の名称】 非水電解質二次電池

(57)【要約】

【課題】 本発明は、高電位での作動が可能な高エネルギー密度を有した非水電解質二次電池を提供することである。

【解決手段】 非水電解質二次電池用の正極活物質として、鉄、コバルト、銅のうち少なくとも1種以上を含む金属ヨウ素酸化合物を用いる。

- 1…電池ケース
- 2…封口板
- 3…集電体
- 4…金属リチウム
- 5…試料槽(正極)
- 6…セパレータ
- 7…ガスケット



【特許請求の範囲】

【請求項1】非水電解質と、正極と、リチウムを吸蔵、放出することができる負極から構成される非水電解質二次電池において、前記正極活物質が金属ヨウ素酸化合物である非水電解質二次電池。

【請求項2】金属ヨウ素酸化合物が鉄、コバルト、銅のうち少なくとも1種以上を含む金属ヨウ素酸化合物であることを特徴とする請求項1記載の非水電解質二次電池。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、非水電解質二次電池の、特に正極材料の改良に関するものである。

【0002】

【従来の技術】非水電解質二次電池は、小型、軽量で、かつ高エネルギー密度を有するため、機器のポータブル化、コードレス化が進む中で、その期待は高まっている。

【0003】従来、非水電解質二次電池用の正極活物質として4V級のLi₂CO₃、LiNiO₂、LiMn₂O₄などのリチウム含有金属酸化物が提案され、一部が実用化されている。一方、負極としては金属リチウム、リチウムを吸蔵・放出することのできる炭素材料などが提案され、炭素材料は実用化されている。

【0004】

【発明が解決しようとする課題】しかしながら、従来の正極活物質では作動電位が概ね4V(リチウム金属負極基準)であり、さらなる高エネルギー密度化を図る上においては十分に対応できない状況にあった。

【0005】そこで、近年では、LiNiVO₄、LiCoVO₄など[G.T.K.Fey, K.S.Wang, and S.M.Yang., J.Power Sources 68 159('97)]、あるいはLiCr_xMn_{2-x}O₄[C.Sigala, D.Guyomard, A.Verbaere, Y.Piffard and M.Tournouxx., Solid state Ionics 81 167 ('95)]、LiNi_xMn_{2-x}O₄[K.Amine, H.tsukamoto, H.Yasuda and Y.Fujita., J.Electrochem.Soc., 143 1607('96)]などにおけるスピネル構造あるいは逆スピネル構造の材料において、4Vを越える高電位作動を示すことが示され、高エネルギー密度化が示唆されている。

【0006】しかしながら、これらの素材では高電位は得られるものの可逆容量は4V級として作動するLi₂CO₃、LiNiO₂、LiMn₂O₄ほど得られず高容量化という観点からは高エネルギー密度化を得ることができないという課題があった。

【0007】本発明は、このような課題を解決するもので、高電位作動で従来よりも高容量な非水電解質二次電池用正極材料を提供するものである。

【0008】

【課題を解決するための手段】これらの問題を解決するために本発明では、非水電解質と、正極と、リチウムを

吸蔵、放出することができる負極から構成される非水電解質二次電池において、上記正極活物質が金属ヨウ素酸化合物である非水電解質二次電池用正極材料を用いるのであり、好ましくは上記金属ヨウ素酸化合物が鉄、コバルト、銅のうち少なくとも1種以上を含む金属ヨウ素酸化合物を正極材料に用いることにしたのである。

【0009】

【発明の実施の形態】金属ヨウ素酸化合物は、ヨウ素1個と酸素3個とからなる4面体構造のヨウ素酸配位種が、金属元素に固有の配位数で配位した結晶学的な基本構造を有している。ヨウ素酸配位種は極めて強い電気陰性度を持ち、配位する金属元素から電子を引き抜く作用が大きい。形式的にはヨウ素酸の負電荷はマイナスの1価であり、金属元素は電荷補償に相当する正電荷とされるが、実質的には化合物中の金属元素の酸化状態は配位種の電気陰性度に応じて連続して変化する種々の状態にあると言われている。つまり同種の金属元素であっても、例えば酸素などのように、配位種の電気陰性度が小さい場合は、金属元素の実質的な酸化状態はそれだけ低くなる。また、金属化合物の電気化学的なリチウムの吸蔵・放出反応における酸化還元電位は、金属元素の実質的な酸化状態に依存しており、その酸化状態が高いと酸化還元電位は高くなる。よって電気陰性度の高いヨウ素酸を配位種とする金属ヨウ素酸化合物の場合には、その酸化還元電位が相応に高くなっているものと考えられる。

【0010】金属ヨウ素酸化合物には、例えば六方晶系のFe(I0₃)₃ (空間群P63に帰属可能)、三方晶系のCo(I0₃)₃ (空間群P3に帰属可能)、単斜晶系の α -Cu(I0₃)₂ (空間群P1211に帰属可能)、 γ -Cu(I0₃)₂ (空間群P121/m1に帰属可能)、正方晶系のZr(I0₃)₄ (空間群P4/n mに帰属可能)、斜方晶系のAgI0₃ (空間群Pbc21に帰属可能)などが知られている。

【0011】本発明者らがこれらの材料で電気化学的な特性を鋭意検討したところ、Fe(I0₃)₃、Co(I0₃)₃、 α -Cu(I0₃)₂、 γ -Cu(I0₃)₂では4Vを越える作動電位を示し、かつ可逆容量成分の体積当たりの容量密度も従来の技術で開示されるものよりも良好になることを見い出した。

【0012】本発明はこのような事実に基づくものであり、以下実施例で詳細に本発明の具体的な内容を説明する。

【0013】

【実施例】(実施例1)図1に本発明の実施例で用いたコイン形非水電解質二次電池の縦断面図を示す。図において1は耐有機電解質性のステンレス鋼板を加工した電池ケース、2は同材料の封口板、3は同材料の集電体で、ケース1の内面にスポット溶接されている。4は金属リチウムで、封口板2の内部に圧着されている。5は本発明の評価用試料正極で、Fe(I0₃)₃、Co(I0₃)₃、 α -

$\text{Cu}(\text{IO}_3)_2$ 、 $\gamma-\text{Cu}(\text{IO}_3)_2$ 、 $\text{Zr}(\text{IO}_3)_4$ 、 AgIO_3 などのヨウ素酸化合物あるいは鉄、コバルト、銅、ジルコニア、銀のうち少なくとも1種以上を含む金属ヨウ素酸化合物を正極活物質にしたものである。極板の作製に当たっては、上記試料85重量部に対し、結着剤としてポリフッ化ビニリデン10重量部と導電剤としてアセチレンブラック5重量部を混合して得られる合剤の所定量を集電体3の上に成形したものである。これらの集電体を80°Cで減圧乾燥した後、電池組立てに用いた。6は微孔性のポリプロピレン製セパレーター、7はポリプロピレン製絶縁ガスケットである。電解液はエチレンカーボネート、ジエチルカーボネートの等体積混合溶媒に溶質として六フッ化リン酸リチウムを1モル/リットルの濃度で溶解して用いた。これらを用いて、寸法が直径20mm、電池総高1.6mmである電池を構成し、充放電サイクル試験*

*試験を行った。評価方法は電流密度0.5mA/cm²の定電流で3~5.1V(リチウム金属負極基準)の範囲で充放電を行い、それを10サイクル繰り返した。なお、本充放電試験に供する前に試料極を3Vまで印加しリチウムを挿入した後通常の充放電試験を行った。(表1)に上記電位走引範囲における平均電位と10サイクル後の放電容量を示した。

【0014】(比較例1)正極活物質に $\text{LiNi}_{1-x}\text{Co}_x\text{VO}_4$ ($0.5 < x < 0.8$)を試料として用い、実施例1に準じて10正極を作製し、それを用いて評価用コイン形非水電解質二次電池を作製した。そして実施例1と同様の評価条件で充放電サイクル試験を行い、平均電位および放電容量を計算した。その結果を(表1)に示した。

【0015】

【表1】

実施例	電位V(Li基準)	10サイクル
	V vs. Li	放電容量 /mA·h·cm ⁻²
$\text{Fe}(\text{IO}_3)_2$	4.49	118
$\text{Co}(\text{IO}_3)_4$	4.54	137
$\alpha-\text{Cu}(\text{IO}_3)_2$	4.37	109
$\gamma-\text{Cu}(\text{IO}_3)_2$	4.43	99
$\text{Zr}(\text{IO}_3)_4$	3.39	91
$\text{Fe}_{0.99}\text{Co}_{0.01}(\text{IO}_3)_2$	4.56	119
比較例 $\text{LiNi}_{1-x}\text{Co}_x\text{VO}_4$ $0.5 < x < 0.8$	4.26	83

【0016】銀のヨウ素酸化合物については、同様の傾向を示し正極候補材料として考えられるが、材料コストの点からは工業的生産の魅力に欠けるため、表からは割愛した。

【0017】(表1)から、本発明の各種ヨウ素酸化合物は非水電解質二次電池の正極材料として機能し、とりわけ鉄、コバルト、銅のうち少なくとも1種以上を含む金属ヨウ素酸化合物は高電位でしかも容量密度に優れることが分かった。また、本発明の正極材料を非水電解質二次電池に用いる場合、負極活物質にはリチウム金属あるいはリチウムを含有する窒化物、酸化物、合金などを用いることができる。

【0018】さらに、本発明の正極材料を非水電解質二次電池に用いる場合、非水電解質の溶媒としては、EC(エチレンカーボネート)、PC(プロピレンカーボネート)、DMC(ジメチルカーボネート)、EMC(エチルメチルカーボネート)、DEC(ジエチルカーボネート)等の鎖状エステル類、アーブチロラクトン等のア

※ーラクトン類、DME(1,2-ジメトキシエタン)、DEE(1,2-ジエトキシエタン)、EME(エトキシメトキシエタン)等の鎖状エーテル類、テトラヒドロフラン等の環状エーテル類、アセトニトリル等のニトリル類等から選ばれた溶媒もしくは2種類以上の混合溶媒を用いることができる。特にEC(エチレンカーボネート)を必須成分として含む混合溶媒を使用することが好適である。そして非水電解質の溶質としては、LiAsF₆、LiPF₆、LiAlC₁₄、LiClO₄、LiC₂SO₃、LiSbF₆、LiSCN、LiCl、LiC₆HSO₃、Li(CF₃SO₂)₂、LiC(CF₃SO₂)₃、C₄F₉SO₃Li等のリチウム塩及びこれらのこれらの混合物を用いることができる。これら溶液系の他に、PVDF、PEO、PAN系などの高分子材料を用いた固体状電解質あるいはゲル状電解質を含む固体-ゲル電解質を用いた電解質においても同様の効果が得られる。

【0019】また、電池の形状に関しては、本実施例で

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はコイン形を用いたが円筒形、角形、その他いかなる形状の電池でも使用できる。

【0020】

【発明の効果】本発明は、正極活物質が金属ヨウ素酸化物である非水電解質二次電池用正極材料を用いるものであり、好ましくは鉄、コバルト、銅のうち少なくとも1種以上を含む金属ヨウ素酸化物を正極材料に用いる非水電解質二次電池であり、高電位での作動が可能な高エネルギー密度を有した電池を提供することができる。

【図面の簡単な説明】

【図1】本発明のコイン形電池の縦断面図

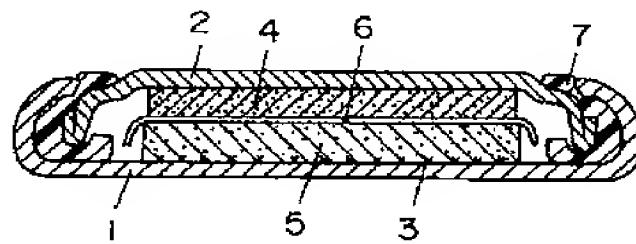
【符号の説明】

- 1 電池ケース
- 2 封口板
- 3 集電体
- 4 金属リチウム
- 5 試料極（本実施例では本発明の正極活物質を記載）
- 6 セパレーター
- 7 ガスケット

10

【図1】

- 1…電池ケース
- 2…封口板
- 3…集電体
- 4…金属リチウム
- 5…試料極(正極)
- 6…セパレーター
- 7…ガスケット



フロントページの続き

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 5H014 AA02 EE10
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 BJ16

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CLAIMS

[Claim(s)]

[Claim 1] The nonaqueous electrolyte rechargeable battery whose aforementioned positive active material is a metal iodine acid compound in nonaqueous electrolyte, a positive electrode, and the nonaqueous electrolyte rechargeable battery that consists of occlusion and a negative electrode which can be emitted in a lithium.

[Claim 2] The nonaqueous electrolyte rechargeable battery according to claim 1 with which a metal iodic-acid compound is characterized by being a metal iodic-acid compound containing at least one or more sorts in iron, cobalt, and copper.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention is the thing especially about improvement of positive-electrode material of a nonaqueous electrolyte rechargeable battery.

[0002]

[Description of the Prior Art] Small, since it is lightweight and has high-energy density, while, as for a nonaqueous electrolyte rechargeable battery, portable-izing of a device and cordless-ization progress, the expectation is growing.

[0003] Conventionally, lithium content metallic oxides, such as LiCoO₂ and LiNiO₂, and LiMn₂O₄, are proposed as a positive active material for nonaqueous electrolyte rechargeable batteries, and the part is put in practical use. [4V class] On the other hand, as a negative electrode, occlusion, the carbon material which can be emitted are proposed in a metal lithium and a lithium, and the carbon material is put in practical use.

[0004]

[Problem(s) to be Solved by the Invention] However, in the conventional positive active material, action potential is 4V (lithium metal negative-electrode criteria) in general, and the situation that further high-energy density-ization was attained upwards and it could not fully respond was suited.

[0005] then -- recent years -- [, such as LiNiVO₄ and LiCoVO₄, -- G. -- T.KFey, KS.Wang, and S.M.Yang., and J.Power Sources 68 159 ('97) -- Or LiCr_xMn_{2-x}O₄[C. Sigala, D.Guyomard, A.Verbaere, Y.Piffard and M.Tournoux., Solid state Ionics 81 167] ('95), In the material of the Spinel structure in LiNi_xMn_{2-x}O₄ [K.Amine, H.tsukamoto, H.Yasuda and Y.Fujita., J.Electrochem.Soc., 143 1607 ('96)] etc., or inverse-spinel structure It is shown that the high potential operation exceeding 4V is shown, and high-energy density-ization is suggested.

[0006] However, for these materials, although high potential was obtained, the reversible capacitance was not obtained LiCoO₂, LiNiO₂, and about 2O₄ LiMn which operate as 4V class, but had the technical problem that high-energy density-ization could not be obtained, from a viewpoint of high-capacity-izing.

[0007] this invention solves such a technical problem and offers a positive-electrode material [high capacity / before] for nonaqueous electrolyte rechargeable batteries by high potential operation.

[0008]

[Means for Solving the Problem] In order to solve these problems, in this invention, the metal iodic-acid compound with which the above-mentioned metal iodic-acid compound contains at least one or more sorts in iron, cobalt, and copper preferably will be used for positive-electrode material using the positive-electrode material for nonaqueous electrolyte rechargeable batteries whose above-mentioned positive active material is a metal iodic-acid compound in nonaqueous electrolyte, a positive electrode, and the nonaqueous electrolyte rechargeable battery that consists of occlusion and a negative electrode which can be emitted in a lithium.

[0009]

[Embodiments of the Invention] The metal iodic-acid compound has the crystallography basic structure which the iodic-acid coordination kind of the tetrahedron structure which consists of one iodine and three oxygen configurated in the coordination number peculiar to a metallic element. An iodic-acid coordination kind has the large operation which has very strong electronegativity and draws out an electron from the metallic element to configurate. Formally, although the negative charge of the iodic acid is univalent [of minus] and a metallic element is made into the positive charge equivalent to charge compensation, it is said that the oxidation state of the metallic element in a compound is in the various states of changing continuously according to the electronegativity of a coordination kind, substantially. That is, even if it is a metallic element of the same kind, when the electronegativity of a coordination kind is small, the substantial oxidation state of a metallic element becomes so low, for example like oxygen. Moreover, it depends for the oxidation reduction potential in the occlusion and release reaction of the electrochemical lithium of metallic compounds on the substantial oxidation state of a metallic element, and if the oxidation state is high, oxidation reduction potential will become high. Therefore, in the case of the metal iodic-acid compound which uses the iodic acid with high electronegativity as a coordination kind, it is thought that the oxidation reduction potential is high suitably.

[0010] To a metal iodic-acid compound, for example, Fe(IO₃)₃ of hexagonal system (attribution in a space group P63 is possible), Co (IO₃)₃ (attribution in a space group P3 is possible) of trigonal system, alpha-Cu 2 (attribution in a space group P1211 is possible) of monoclinic system (IO₃), As for gamma-Cu (IO₃)₂, AgIO₃ (attribution in a space group Pbc21 is possible) of (attribution in space groups P121/m1 is possible), Zr (IO₃)₄ (attribution in space groups P4/nZ is possible) of tetragonal system, and orthorhombic system etc. is known.

[0011] When this invention persons examined the electrochemical property wholeheartedly with such material, in Fe (IO₃)₃, Co (IO₃)₃, alpha-Cu (IO₃)₂, and gamma-Cu (IO₃)₂, the bird clapper was found out better than the thing which shows the action potential exceeding 4V and by which the capacity density per volume of a reversible-capacitance component is also indicated by the Prior art.

[0012] this invention explains the concrete content of this invention in detail in the example below based on such a fact.

[0013]

[Example] (Example 1) Drawing of longitudinal section of the coin form nonaqueous electrolyte rechargeable battery used for drawing 1 in the example of this invention is shown. The cell case into which 1 processed the stainless steel plate of organic-electrolyte-proof nature in

drawing, and 2 are the charge collectors of this material, and spot welding of the obturation board of this material and 3 is carried out to the inside of a case 1. 4 is a metal lithium and is stuck to the interior of the obturation board 2 by pressure. 5 is the sample positive electrode for evaluation of this invention, and makes iodic-acid compounds, such as $\text{Fe}(\text{IO}_3)_3$, $\text{Co}(\text{IO}_3)_3$, $\alpha\text{-Cu}(\text{IO}_3)_2$, $\gamma\text{-Cu}(\text{IO}_3)_2$, and $\text{Zr}(\text{IO}_3)_4$, AgIO_3 , or iron, cobalt, copper, a zirconium, and the metal iodic-acid compound containing at least one or more sorts in silver a positive active material. If in charge of production of a plate, the specified quantity of the mixture which mixes the acetylene black 5 weight section as the polyvinylidene-fluoride 10 weight section and an electric conduction agent as a binder, and is obtained is fabricated on a charge collector 3 to the above-mentioned sample 85 weight section. It used for the cell assembly, after carrying out reduced pressure drying of these charge collectors at 80 degrees C. The separator made from polypropylene of microporosity [6] and 7 are the insulating gaskets made from polypropylene. The electrolytic solution dissolved and used the 6 fluoride [phosphoric-acid] lithium for volume mixed solvents, such as ethylene carbonate and diethyl carbonate, by the concentration of one mol/l. as a solute. Using these, the cell whose sizes are the diameter of 20mm and 1.6mm of cell total amounts was constituted, and the charge-and-discharge cycle examination was performed. The evaluation method performs charge and discharge in 3-5.1V (lithium metal negative-electrode criteria) by the constant current of current density 0.5 mA/cm², and is 10 cycle ***** about it. In addition, before presenting this charge and discharge test, after impressing the sample pole to 3V and inserting a lithium, the usual charge and discharge test was performed. The service capacity after the average potential in the above-mentioned potential **** range and 10 cycles was shown in (Table 1).
 [0014] (Example 1 of comparison) $\text{LiNi}_{1-x}\text{Co}_x\text{VO}_4$ ($0.5 < x < 0.8$) was used for the positive active material as a sample, the positive electrode was produced according to the example 1, and the coin form nonaqueous electrolyte rechargeable battery for evaluation was produced using it. And the charge-and-discharge cycle examination was performed on the same evaluation conditions as an example 1, and average potential and service capacity were calculated. The result was shown in (Table 1).

[0015]

[Table 1]

実施例	電位V(Li基準)	10サイクル
	V vs. Li	放電容量 /mAh·cm ⁻²
$\text{Fe}(\text{IO}_3)_3$	4.49	118
$\text{Co}(\text{IO}_3)_3$	4.54	137
$\alpha\text{-Cu}(\text{IO}_3)_2$	4.37	109
$\gamma\text{-Cu}(\text{IO}_3)_2$	4.43	99
$\text{Zr}(\text{IO}_3)_4$	3.39	91
$\text{Fe}_{0.5}\text{Co}_{0.5}(\text{IO}_3)_3$	4.56	119
比較例 $\text{LiNi}_{1-x}\text{Co}_x\text{VO}_4$ $0.5 < x < 0.8$	4.26	83

[0016] Although the same inclination was shown and the silver iodic-acid compound was considered as a positive-electrode candidate material, since the charm of industrial production was missing, from the point of material cost, it omitted from the table.

[0017] The various iodic-acid compounds of this invention functioned as a positive-electrode material of a nonaqueous electrolyte rechargeable battery, and (Table 1) showed that iron, cobalt, and the metal iodic-acid compound containing at least one or more sorts in copper were moreover especially excellent in capacity density with high potential. Moreover, when using the positive-electrode material of this invention for a nonaqueous electrolyte rechargeable battery, the nitride containing a lithium metal or RICHUMU **, an oxide, an alloy, etc. can be used for a negative-electrode active material.

[0018] When using the positive-electrode material of this invention for a nonaqueous electrolyte rechargeable battery, furthermore, as a solvent of nonaqueous electrolyte EC (ethylene carbonate), PC (propylene carbonate), DMC (dimethyl carbonate), EMC (ethyl methyl carbonate), gamma-lactone, such as chain-like ester, such as DEC (diethyl carbonate), and gamma-butyrolactone, DME (1, 2-dimethoxyethane), The solvent chosen from nitril, such as cyclic ether, such as chain-like ether, such as DEE (1, 2-diethoxy ethane) and EME (ethoxy methoxyethane), and a tetrahydrofuran, and an acetonitrile, or two or more kinds of mixed solvents can be used. It is suitable to use the mixed solvent which contains especially EC (ethylene carbonate) as an indispensable component. and -- as the solute of nonaqueous electrolyte -- LiAsF_6 , LiPF_6 , LiAlCl_4 , LiClO_4 , and LiCF_3 -- lithium salt and such mixture of such, such as SO_3 , LiSbF_6 , LiSCN , LiCl , LiC_6HSO_3 , $\text{Li}(\text{CF}_3\text{SO}_2)_2$ and $\text{LiC}(\text{CF}_3\text{SO}_2)_3$, and $\text{C}_4\text{F}_9\text{SO}_3\text{Li}$, can be used. The same effect is acquired also in the electrolyte using the solid-state-gel electrolyte containing the solid-state-like electrolyte or gel electrolyte which used polymeric materials other than these solution system, such as a PVDF, PEO, and PAN system.

[0019] Moreover, about the configuration of a cell, by this example, although the coin form was used, it can be used by the cell of a cylindrical shape, a square shape, and any other configurations.

[0020]

[Effect of the Invention] this invention is a nonaqueous electrolyte rechargeable battery which uses for positive-electrode material the metal iodic-acid compound with which at least one or more sorts in iron, cobalt, and copper are preferably included using the positive-electrode

material for nonaqueous electrolyte rechargeable batteries whose positive active material is a metal iodic-acid compound, and can offer a cell with the high-energy density in which the operation with high potential is possible.

[Translation done.]